ADAPTORS FOR THE AEROGRAPH PYROLYZER

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Users of the Aerograph Model A-425 Pyrolyzer Accessory will no doubt agree that it would be highly desirable to be able to inject standard samples onto the same column as that used for pyrolysis, the purpose, of course, being the identification of unknown peaks in the pyrogram. This can be done, but it requires disconnection of the auxiliary carrier gas line and removal of the pyrolytic chamber before a septum and nut can be added to the injection port. A more convenient method is to use an adaptor such as that illustrated in Figure 1A. This device threads into the pyrolysis chamber, accepts a standard septum and is capped with a regular injection-port nut or a collector nut. Injections can now be made in the usual manner with the needle inserted beyond the auxiliary carrier gas entry line of the pyrolysis chamber to assure adequate pickup of the sample. The adaptor may be kept in place even when pyrolytic studies are not being made, thus permitting normal use of the column without removal of the pyrolysis chamber.

Another adaptor that may be quite useful to the pyrolysis worker is that shown in Figure 1B. This device is nothing more than a solid plug that threads into the pyrolysis chamber. It provides a positive closure to the system, allowing the carrier gas to flow through the column in the normal manner, whenever the micro-dot connector is not in place.

Figure 2 is an exploded photographic view of both adaptors. These units increase the convenience and versatility of the Aerograph Pyrolyzer.

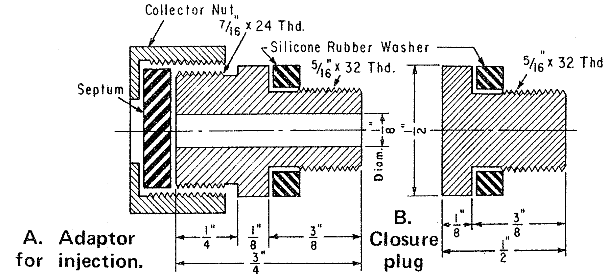


Fig 1. Construction diagram of adaptors. All dimensions are in inches; material is stainless steel.

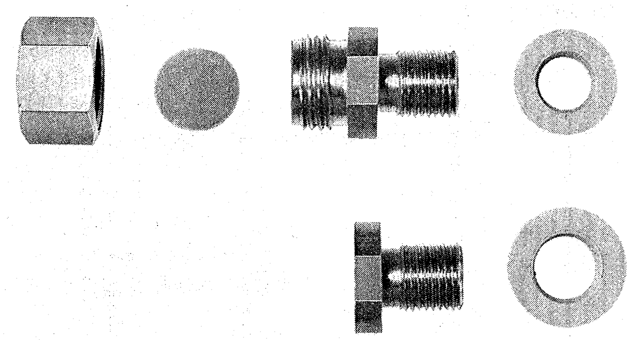


Fig. 2. Exploded view of adaptors.

ACKNOWLEDGEMENT

The author acknowledges the fine work of Mr. William J. Eglinton in the construction of the adaptor parts.

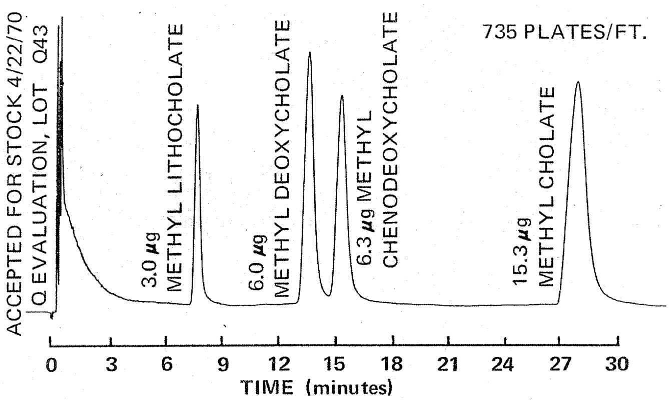
Note: Applied Science Laboratories does not manufacture or sell the above adaptors. All correspondence concerning them should be directed to the author.

GAS-CHROM Q – JUST A REMINDER

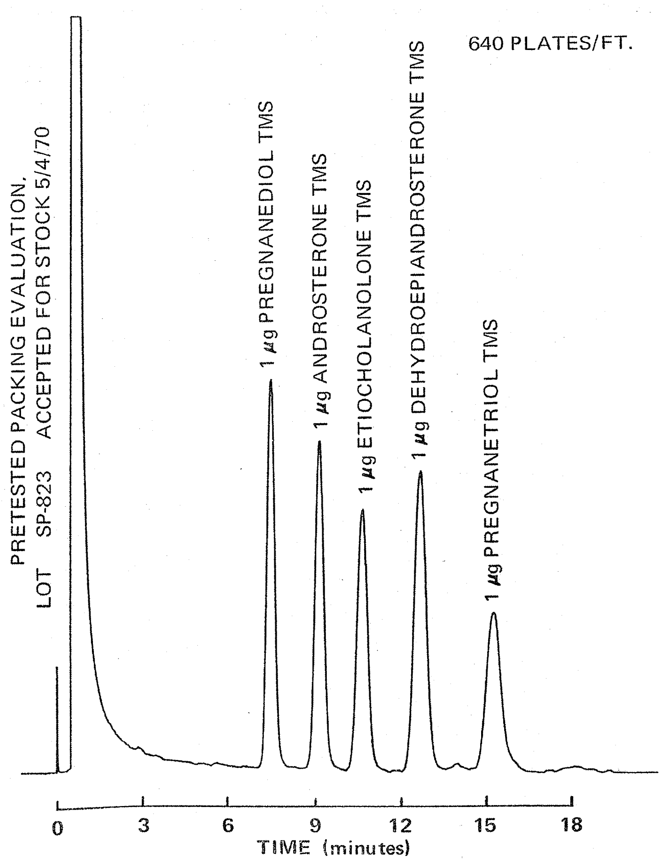
GAS-CHROM Q is the highest quality silane-treated diatomaceous earth support available for gas chromatography. We could say 5000 words on the subject, but we believe in the old adage that one chromatogram – oops, one picture – is worth a thousand words. So – here's a sampling of some quality control analyses of recent batches of GAS-CHROM Q. Incidentally, all columns were 6ft. x 4mm. I.D. glass.

GAS-CHROM Q

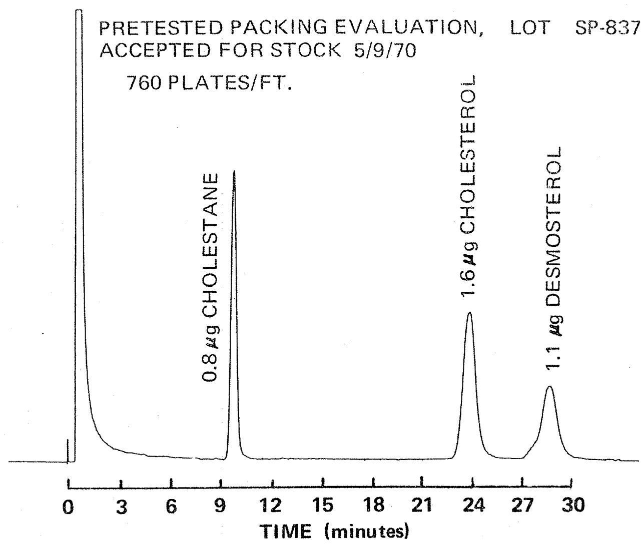
Mesh	Cat. No.	Price/100 g
60/80	02001	$30.00
80/100	02002	35.00
100/120	02003	26.00



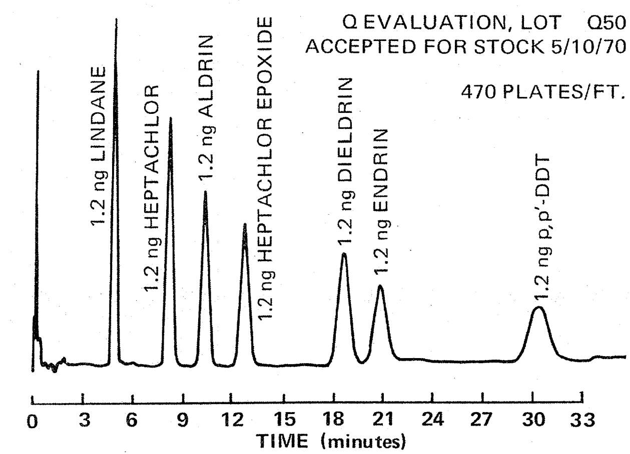
GAS-CHROM Q, 100/120 mesh, coated with 3% QF-1. Column temperature: 250°C. Detector: FID at 9×10^{-10}AFS. N_2 flow rate: 109 ml/min. at 30 psig.



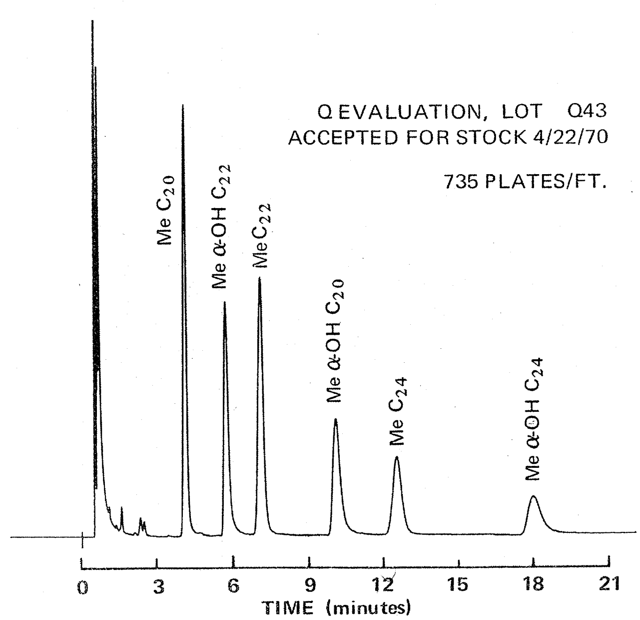
GAS-CHROM Q, 100/120 mesh, coated with 3% OV-225. Column temperature: 230°C. Detector: FID at 9×10^{-10}AFS. N_2 flow rate: 38 ml/min. at 13 psig.



GAS-CHROM Q, 100/120 mesh, coated with 3% OV-17. Column temperature: 250°C. Detector: FID at 5×10^{-10}AFS. N_2 flow rate: 27 ml/min. at 13 psig.



GAS-CHROM Q, 100/120 mesh, coated with 10% DC 200. Column temperature: 200°C. Detector: FID at 9×10^{-10}AFS. N_2 flow rate: 85 ml/min. at 25 psig.



GAS-CHROM Q, 100/120 mesh, coated with 3% JXR. Column temperature: 225°C. Detector: FID at 9×10^{-10}AFS. N_2 flow rate: 35 ml/min. at 17 psig.

MONOMOLECULAR LAYERS AND LIGHT

An interesting article by Karl H. Drexhage, published in *Scientific American,* Vol. 222, No. 3, (March, 1970), has been brought to our attention. Multilayer systems of long-chain fatty-acid molecules and fluorescent dye molecules have been used to study the structure of light waves, revealing a close analogy between a radio antenna and a light-emitting molecule.

It was reported that even though visible light is the most familiar form of electromagnetic radiation, some aspects of the generation and propagation of light waves have not been as carefully studied as microwaves or radio waves. The reason for this is traced to the short wavelength of visible light, which is small compared with the dimensions of most physical apparatus.

Recently a new investigative technique, developed by Karl H. Drexhage and his colleagues at the University of Marburg in Germany, makes use of thin layers of matter to probe the structure of various light waves. Among other things, they found that when an excited dye molecule emits light by the process of fluorescence, its behavior is remarkably analogous to that of an antenna emitting radio waves.

The monomolecular-layer systems are built up by dropping a benzene solution of a long-chain fatty-acid on the surface of water in a small trough. The volatile solvent evaporates quickly, leaving the molecules of the water-insoluble fatty acid at the surface with their hydrophilic groups sticking into the water. By adding more fatty acid solution it is possible to cover the entire water solution with a monomolecular film of the fatty acid. A small force is applied to exert pressure on the film thus causing the fatty acid molecules to stand upright at the surface of the water. The thickness of the monomolecular film is simply the length of the fatty acid which was usually partly converted to its cadmium salt, (CdC_{20}) by adding a small amount of cadmium chloride (CdC_{12}) to improve the stability of the layers.

A clean glass slide is lowered through the water surface and as the slide is withdrawn the hydrophilic carboxyl groups adhere to the glass, and a monomolecular layer of the fatty acid is attached to both sides of the slide. The glass emerges completely dry and its surface is now hydrophobic, because the terminal methyl groups of the fatty acid molecules are pointing outward. This procedure can be repeated so that a multilayer system is built up. The entire procedure is quite simple. All one needs are pure chemicals, a trough with a float, and a dipping device. The researchers using this method have been able to deposit more than 500 monomolecular fatty acid layers of excellent optical quality.

Various methods have been used to check whether the layers are built up smoothly and reproducibly, one an electrical method and the other an optical method.

Besides fatty acids other substances have been used successfully for the preparation of multilayer systems. For the studies that would absorb and emit visible light, dyelike substances were needed. Suitable dye substances were developed by chemical syntheses. However, it was found that by adding arachidic acid to the dye solution the monomolecular dye layers proved to be more stable against diffusion.

Two specific light fields that were investigated with this technique are the standing light wave in front of a mirror and the boundary wave accompanying total reflection. In both of these experiments a monomolecular layer of a fluorescent dye was used as a probe for the electric field of the light wave. One positions the dye layer inside the field pattern by varying the number of CdC_{20} layers. Then by measuring the intensity of the fluoresence one can probe the structure of the light field quantitatively. Because the monomolecular layersare very thin compared with the wavelength of visible light, a spatial resolution difficult to obtain with other methods is readily achieved.

The systems of monomolecular layers can be regarded as artificial crystals in which one can choose the composition within wide limits. It is reasonable to envision numerous applications of this technique in other areas of science.

It was reported that fatty acid layer systems grown from a solution containing metal ions such as barium and lead, exhibit a spacing of about 50 angstroms between the planes of the heavy metal ions. These systems have been used successfully as analyzers for soft x-rays and may be useful in future lasers.

Many natural organic compounds such as cholesterol are also suitable for the preparation of layer systems. Research involving these artificial crystals may provide a fresh insight into some life processes.

Waters Associates has just announced a price increase for the column packing materials it manufactures, listed below.

NEW PRICES

PORAPAK

Porapak	Catalog Number 50/80 mesh	80/100 mesh	100/120 mesh	Price
Q	05922	05923	05924	$17.00/75 cc
P	05912	05913	05914	19.00/75 cc
R	05932	05933	05934	19.00/75 cc
S	05942	05943	05944	19.00/75 cc
T	05952	05953	05954	19.00/75 cc
N	05902	05903	05904	19.00/75 cc
QS	05962	05963	05964	22.00/75 cc
PS	05972	05973	05974	25.00/75 cc

PORASIL

Price: $18.00/75 cc

Porasil	Surface Area m^2/g	Pore Diameter Angstroms	Catalog No.
A	480	< 100	05891
B	200	100 - 200	05892
C	50	200 - 400	05893
D	25	400 - 800	05894
E	4	800 - 1500	05895
F	1.5	> 1500	05896

DURAPAK

Durapak Type	Cat. No.	Price
OPN/ PORASIL C	05998	$49.00/75 c
Carbowax 400/ PORASIL C	05996	49.00/75 c
n-Octane/PORASIL C	05994	49.00/75 c
Carbowax 400/PORASIL S	05997	49.00/75 c
Phenyl Isocyanate/PORASIL C	05999	49.00/75 c
Carbowax 4000/PORASIL S	06000	49.00/75 c

OV-61, A New Silicone Phase

Several years ago General Electric released samples of the stationary phase XE-61, a 33% phenyl substituted silicone. Since that time they have not marketed the phase and apparently have no intention of doing so.

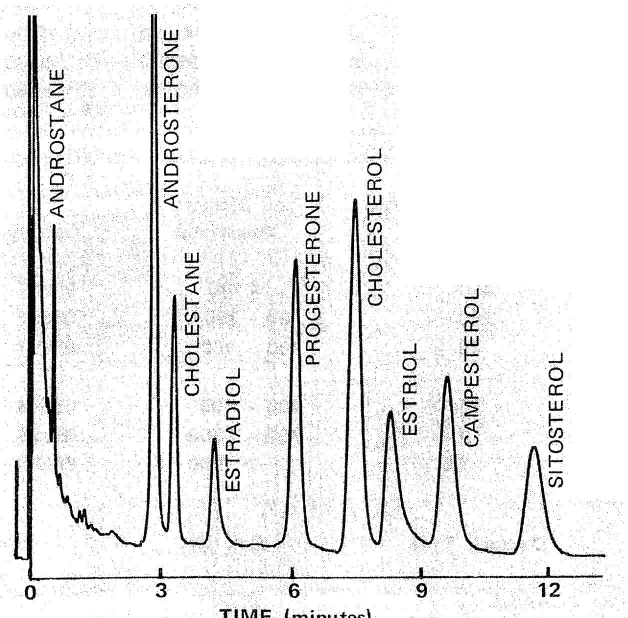
Now Applied Science Laboratories and Ohio Valley Specialty Chemical Company have teamed up to manufacture and test OV-61, also a 33% phenyl substituted silicone. When we compared this phase to a sample of XE-61, we found that we could not detect any significant differences between the two with respect to their GC properties. This was pleasant news to us, because we had previously considered XE-61 to be an outstanding phase and regretted that GE had not made it commercially available. OV-61 seems to be the ideal replacement.

OV-61 possesses a selectivity for steroids intermediate between OV-1 and OV-17, as can be seen from the retention data shown in the table below.

	OV-1 (0% phenyl)	OV-61 (33% phenyl)	OV-17 (50% phenyl)
Cholestane	1.00	1.00	1.00
α-Coprostanol	1.55	1.92	1.97
β-Cholestanol	1.84	2.16	2.27
Desmosterol	-------	2.16	2.27
Cholesterol	1.68	2.15	2.26
Campesterol	2.11	2.74	2.88
Stigmasterol	2.28	2.92	3.09
Sitosterol	2.69	3.31	3.48

Columns: 6 ft. x 4 mm I.D. glass U-tubes packed with 3% phase on 100/120 mesh GAS-CHROM Q. Column temperature: 270°C. Detector: Flame ionization at 5 x 10^{-10} AFS. N_2 flow rate: 40 ml/min.

We prepared an artificial mixture of several assorted steroids to display the general performance of the new phase. Analysis of all the steroids looks good except for estriol, which, as expected, tails badly. Our chromatogram is shown below.

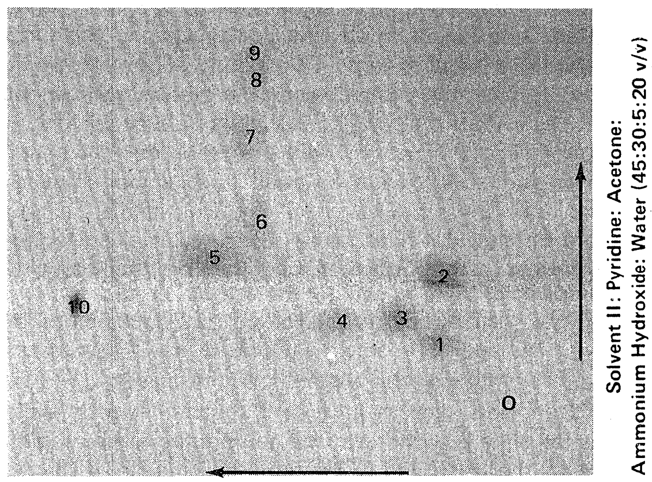


Separation of steroids on OV-61. Column: 6 ft. x 4 mm I.D. glass U-tube packed with 3% OV-61 on 100/120 mesh GAS-CHROM Q. Column temperature: 270°C. Detector: Flame ionization at 1 x 10^{-9} AFS. N_2 flow rate: 50 ml/min.

OV-61	Cat. No. 08254	Price: $ 25.00/10 g

AMINO ACID SEPARATION ON MICROCRYSTALLINE CELLULOSE

The advent of paper chromatography was a giant step forward in amino acid analysis. Another giant step was the invention of the automatic amino acid analyzer, based on ion exchange resins. The difference in expense between the two methods is considerable. However, there is a third technique for analysis of amino acids that is only slightly more expensive than paper chromatography yet gives you the speed of the automatic analyzer (although, unfortunately, not the automatic readout of results). This third method is two dimensional TLC of amino acids on microcrystalline cellulose. Why microcrystalline cellulose, rather than the usual silica gel TLC adsorbent? Because amino acid Rf values are much higher on cellulose, so you obtain more complete separations. On microcrystalline cellulose two dimensional analyses are complete within a few hours - it doesn't take days, as with ordinary paper chromatography. Spots are compact and clear-cut. And it is even easier to prepare a good microcrystalline cellulose TLC plate than it is to produce a good silica gel coated plate. The photograph will give you some idea of the excellent results obtainable.



Solvent I: Isopropanol: Formic Acid: Water (75:12.5:12.5 v/v)

Separation of 10 amino acids on Microcrystalline Cellulose. Amino acids (1.6 µg of each acid): 1. L-Cystine; 2. L-Glutamic acid; 3. L-Lysine; 4. L-Histidine; 5. L-Threonine; 6. L-Proline; 7. L-Tryptophan; 8. L-Phenylalanine. 9. L-Leucine; 10. L-Asparagine. Detection: Ninhydrin spray reagent. First solvent: Isopropanol/formic acid/water (75/12.5/12.5, v/v). Second solvent: Pyridine/acetone/ammonium hydroxide/water (45/30/5/20, v/v).

	Cat. No.	Price
Microcrystalline Cellulose	16271	$9.00/lb.

TWO NEW DURAPAKS

Two new Durapak packings for GC are now available. Like the other Durapaks previously offered, they consist of a solid support to which a liquid phase has been permanently bonded. Phenyl Isocyanate/Porasil C is intended specifically for separation of C_1-C_3 hydrocarbons. Its upper temperature limit is 60°C. Carbowax 4000/Porasil S is more polar than other Durapaks and therefore is recommended for the separation of polar compounds. Its upper temperature limit is 200°C.

Durapak Type	Cat. No.	Price
Phenyl Isocyanate/Porasil C	05999	$49.00/75 cc
Carbowax 4000/Porasil S	06000	49.00/75 cc

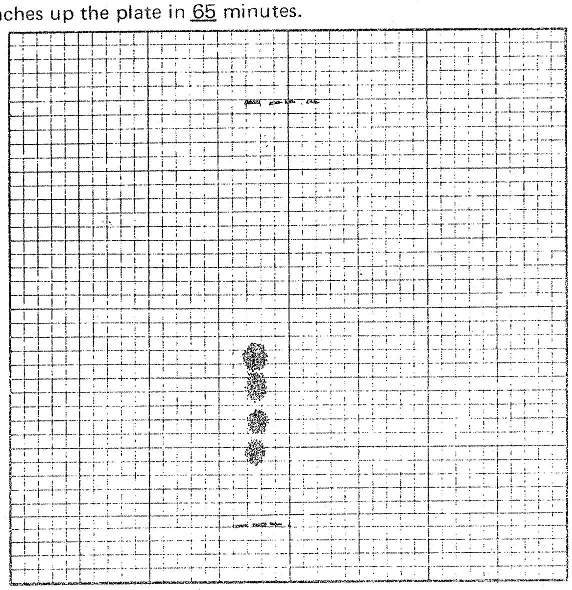
PRETESTED ADSORBOSIL-1

QUALITY CHECK CERTIFICATE

ADSORBOSIL-1

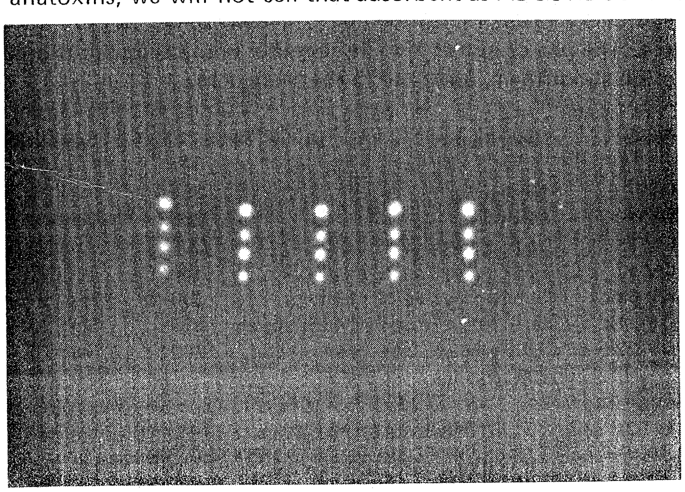
Lot 846-056
Date 320-87
Batch 5/7/70

Test mixture (Aflatoxins): (in descending order) B_1, B_2, G_1, and G_2.
Solvent system: Chloroform/acetone/water, 88/12/1.5 (v/v).
Detecting agent: Long wavelength UV light.
Prepare TLC plates by mixing 100 gms. of the adsorbent with 117 ml. distilled water. Air dry the TLC plates and activate them for 30 minutes before use at 110°C. Cool the activated plates to room temperature and spot the mixture. Develop the plates in a covered and saturated tank. The solvent front moves at 24°C., 7 inches up the plate in 65 minutes.



Actual copy of the separation is kept on file for reference.

The TLC separation of aflatoxins is not easy – it requires an adsorbent that meets certain strict specifications. Because ADSORBOSIL-1 is widely used for aflatoxin analysis (a typical separation is shown below), we selected a mixture of aflatoxins B_1, B_2, G_1, and G_2 as our pretesting standard for this adsorbent. If we find from our quality check procedure that a batch of adsorbent is ineffective for separating these aflatoxins, we will not sell that adsorbent as ADSORBOSIL-1.



Separation of aflatoxins on ADSORBOSIL-1. From top to bottom: Aflatoxins B_1, B_2, G_1, and G_2. Solvent system and other conditions same as on quality check certificate.

You can see from the sample quality check certificate that we furnish you with the exact conditions for the standard separation. There is no room for ambiguity. By using our stated conditions you must be able to duplicate the separation shown on the certificate. Even though you are not interested in analyzing aflatoxins, the certificate is your guarantee that the quality of the ADSORBOSIL-1 you receive will be consistently high.

All ADSORBOSILS, from 1 through 5, are now pretested. Our phosphorescent adsorbents are also prepared from pretested ADSORBOSILS.

PRETESTED ADSORBOSILS FOR TLC

	Cat. No.	Price
ADSORBOSIL-1 (pretested with aflatoxin mixture)	16002	$12.00/1 lb*
ADSORBOSIL-2 (pretested with neutral lipid mixture)	16012	$12.00/1 lb*
ADSORBOSIL-3 (pretested with carbohydrate mixture)	16232	$12.00/1 lb*
ADSORBOSIL-4 (pretested with steroid mixture)	16235	$12.00/250 g $20.00/500 g
ADSORBOSIL-5 (pretested with a phospholipid mixture)	16238	$12.00/1 lb*

* If 4 or more lbs. are ordered, the price is $11.00/1 lb.

Chromosorb Century Series Porous Polymer Beads -- A New Kit

All Johns Manville porous polymer beads (Chromosorbs 101 through 105) are available from Applied Science. Each polymer has its own type of selectivity and can be used for various special applications. In addition, although each packing was designed to separate a definite group of compounds, complex mixtures can be analyzed by careful selection of the polymer.

Applications suggested by the manufacturer are listed below.

Packing	Application	Bulletin
Chromosorb 101	Low molecular weight acids	FF177
Chromosorb 102	Alcohols	FF157A
Chromosorb 103	Amines	FF181
Chromosorb 104	Gases	FF189
Chromosorb 105	Formaldehyde, gases, and low boiling organics (below 200°C).	FF194A

Because of the wide range of applications covered by Chromosorbs 101 through 105, we have assembled a kit containing 20 g of each polymer (the 60/80 mesh size), plus all five bulletins listed in the above table. The kit allows you to try the various types without having to spend a large amount of money (if you bought the regular size of all five packings it would cost you about $400).

Chromosorb Century Polymer Kit Cat. No. 04870 Price: $85.00

New Merck TLC Adsorbents

For your convenience we are now stocking the TLC adsorbents listed below. All are manufactured by Merck (Darmstadt).

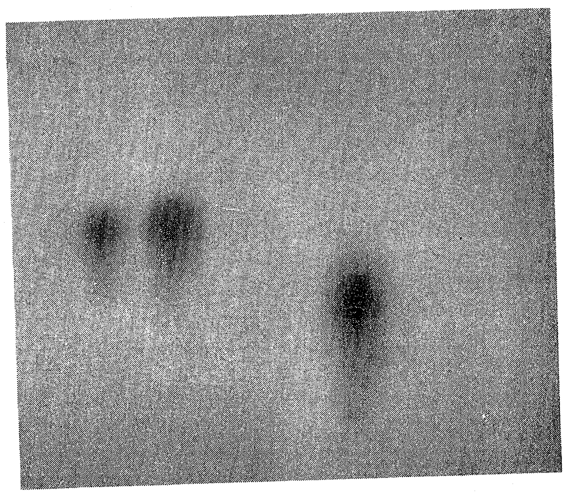
Adsorbent	Cat. No.	Price
Silica Gel G	16310	$10.00/500 g
Silica Gel H	16311	10.90/500 g
Silica Gel HR	16312	19.30/500 g
Silica Gel GF-254	16313	10.90/500 g
Silica Gel HF-254	16314	11.70/500 g
PEI-Cellulose	16315	22.50/500 g
Kieselguhr G	16316	10.00/500 g
Polyamide Powder	16317	30.00/500 g

were lost. We believe that this reagent can be used profitably for analysis of short-chain fatty acids obtained from butterfat, oxidative ozonolysis of fatty acids, and medium-chain triglycerides. The presence of chlorine in the molecule makes 2-chloroethyl derivatives particularly attractive for use with an electron capture detector, which can detect amounts in the nanogram range. For still another application, see the paper by Woodham, Mitchell, and Loftis, summarized in Abstracts, 158th meeting, Am. Chem. Soc., New York, 1969.

PURITY OF REAGENTS

The reagent is prepared with great care from freshly distilled 2-chloroethanol and 99.5% pure boron trichloride.

Boron Trichloride–2-Chloroethanol (10% W/V)
Cat. No. 18052 Price: $20.00



Chromatogram of a fatty acid and its 2-chloroethyl reaction product showing the completeness of esterification. From left to right: 1. Reaction product of fatty acid with 5% BCl_3 in 2-chloroethanol; 2. Reaction product of fatty acid with 10% BCl_3 in 2-chloroethanol; 3. Free fatty acid. Adsorbent: ADSORBOSIL-5 PREKOTE. Solvent system: Hexane/ethyl ether/acetic acid, 90/10/1 (v/v). Detection: Iodine vapor.

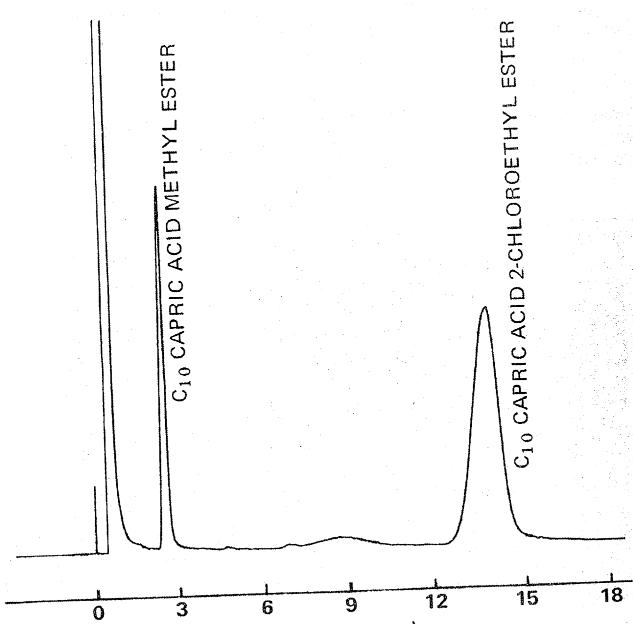


figure 2.

Comparison of retention times of methyl derivatives and 2-chloroethyl derivatives of capric acid. Column: 6 ft. x 4 mm I.D. glass packed with 10% EGSS-X on 100/120 mesh GAS-CHROM P. Column temperature: 147°C. Detector: Flame ionization at 1×10^{-8} AFS.

Instant Methanolic HCl Kit

A very useful reagent for preparing methyl esters of fatty acids is methanolic HCl. References to it are numerous in the literature. However, preparation and storage have always been a problem. First of all, you need a tank of HCl gas, along with the associated valves and accessories. Then you have to prepare the reagent frequently, because within a month methanolic HCl changes to methyl chloride. In addition, a tank of HCl gas represents a considerable laboratory hazard.

Now that's all changed. We put together a kit containing ampoules of acetyl chloride and a bottle of LIPOPURE methanol. Freshly distilled acetyl chloride reacts with anhydrous LIPOPURE methanol to produce hydrogen chloride and methyl acetate. The hydrogen chloride then dissolves in the methanol to yield methanolic HCl.

$$CH_3\text{-}OH + CH_3\text{-}\overset{O}{\overset{\|}{C}}\text{-}Cl \longrightarrow CH_3\text{-}O\text{-}\overset{O}{\overset{\|}{C}}\text{-}CH_3 + HCl\uparrow$$

Methyl acetate formed as a by-product of this reaction does not interfere with the esterification of fatty acids and other lipid samples, or with the GC analysis of the products. With our new kit you can make use of the above reaction to prepare fresh methanolic HCl whenever you want.

The kit contains one pint of anhydrous LIPOPURE methanol and five 5 ml ampoules of redistilled acetyl chloride. All you have to do in order to generate a 3% w/w (2.5% w/v) solution of methanolic HCl is add the contents of a 5 ml ampoule of acetyl chloride to 100 ml of methanol. The reaction is quite vigorous so the acetyl chloride should be added dropwise. The reagent is ready for use 15 minutes after all the acetyl chloride has been added and will remain stable for about one week.

Methanolic HCl reagent has never before been so easy to prepare. If you want to make methyl esthers for GC, you should have this kit.

Instant Methanolic HCl Kit Cat. No. 18053 Price: $18.00/kit

Corning Chromatography Test Kit

In our March-April Newsletter we announced a test kit for permeation chromatography which contained five 50 cc samples of Corning's new controlled-pore glass (CPG-10). Corning has since expanded the kit to contain an additional three 50 cc samples, making a total of eight CPG-10 packings, with pore sizes ranging from 75 through 2000 Angstroms.

The new kit will be available after July 1, 1970, and will contain: 1. A 50 cc analytical column; 2. Eight 50 cc samples of controlled-pore glass (75 through 2000 Angstroms); 3. All packing information and data sheets presently available on the use of CPG-10 packings. This kit will prove an excellent investment for the analyst who would like to try something better than gels for permeation chromatography.

Among the advantages of controlled-pore glass over the gels presently in use are: 1. Water elution may be used; 2. Swelling is non-existent; 3. High pressure may be used without fear of the gel collapsing; 4. Pore size is independent of pressure and solvent; 5. Packing of the column is simple.

Permeation Chromatography Kit Cat. No. 05581 Price: $95.00

CHROMOSORB PRICE INCREASES

On August 1, 1970, all prices of Chromosorb supports will be increased by approximately 10%. All orders received before August 1 will be shipped and billed at the present price. Order now to get this savings.

New Esterification Reagent

BCl_3–2–Chloroethanol (10% w/v)

Because of their volatility and solubility in water, short-chain fatty acid methyl esters are not easy to analyze. In comparison, 2-chloroethyl esters of short-chain fatty acids possess three advantages: (1) they are less volatile, (2) they are less soluble, (3) they are sensitive to electron capture detectors. We wanted to market this novel reagent over a year ago but we waited until now so we could fully evaluate the conditions of use and study possible side reactions.

PREPARATION

To prove the effectiveness of our BCl_3–2-chloroethanol reagent, we placed in a test tube approximately 10 mg of a known quantitative mixture of short-chain fatty acids and added 10 ml of BCl_3–2-chloroethanol. The test tube was immersed in a boiling water bath for 30 minutes, after which 10 ml of ice water was added. The 2-chloroethyl esters were extracted with three 10 ml portions of petroleum ether (b.p. 30-60°C). The organic layers were combined and passed through a filter paper containing anhydrous sodium sulfate. Finally the petroleum ether was concentrated to two ml by evaporation with nitrogen on a water bath.

QUANTITATION

Analysis of the 2-chloroethyl esters was carried out with a 6 ft. x 4 mm I.D. gas chromatography column containing 10% EGSS-X on 80/100 mesh GAS-CHROM P, at 144°C, using a flame ionization detector. (See figure 1.) When the peak areas of the chromatogram were determined, the following table was obtained:

Fatty Acid	Wt. % in Mixture	Area % from Chromatogram
C_6 Caproic	20.0	19.5
C_7 n-Heptanoic	20.0	19.4
C_8 Caprylic	20.0	20.8
C_9 n-Nonanoic	20.0	19.0
C_{10} Capric	20.0	21.6

The above data indicate that the quantitation is reasonably accurate.

COMPLETENESS OF REACTION WITH 2-CHLOROETHANOL

The reaction of 2-chloroethanol with fatty acids appears to go to completion. TLC analysis of the reaction products on an ADSORBOSIL-5 PREKOTE did not reveal the presence of any unreacted fatty acids or any byproducts of the esterification (see photo below).

ADVANTAGES

Volatile short-chain fatty acids can easily be converted to 2-chloroethyl esters. Even when an excess of water was deliberately added during extraction no 2-chloroethyl esters

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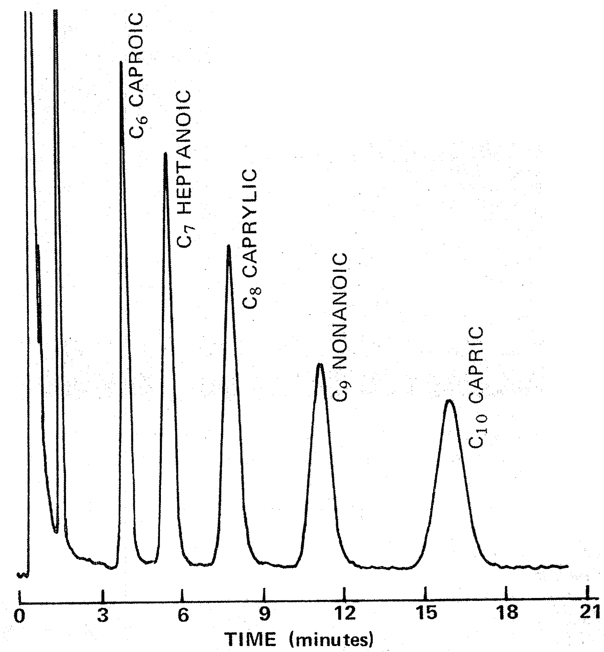

figure 1.

Separation of 2-chloroethyl derivatives of short-chain fatty acids, showing completeness of quantitation. See table in article. Column conditions are given in article.